"A MANUAL FOR THE ANALYSIS OF BUTYLTINS IN ENVIRONMENTAL SAMPLES"
PREPARED FOR THE VIRGINIA DEPARTMENT OF ENVIRONMENTAL QUALITY NOVEMBER 1996

Facility Name:_____VELAP ID_____

Assessor Name:_____ Analyst Name:_____ Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
Were laboratory blanks analyzed with each batch of samples?	p. 7				
Were replicates analyzed at a rate of 10% of samples?	p. 7				
Was every new lot of Grignard reagent tested for butyltins?	p. 22				
Were aqueous samples acidified to a pH of less than 2 with HCl and stored at 4°C for not longer than 13 weeks prior to analysis?	p. 24				
Were sediments kept frozen until analysis?	p. 24				
Analysis in Water Samples					
Were samples brought to room temperature prior to extraction?	p. 8				
Was the surrogate standard (tripentyltin chloride in ethanol) brought up to room temperature before use?	p. 8				
Was all glassware rinsed with hexane prior to use?	p. 8				
Where sample sites were known or suspected to have low analyte concentrations, were 2-Liters of sample collected?	p. 8				
Was the pH of all samples adjusted to 2 with HCl prior to extraction?	p. 9				
Were QA/QC samples prepared with deionized water at the same volume as the volume of samples?	p. 9				
Was TPT internal standard added to all samples, including blank, prior to extraction with hexane/tropolone mixture? (TPT internal standard added prior to extraction, and TBT internal standard added after extraction.)	p. 9				
Was the hexane/tropolone mixture composed of 0.2% tropolone dissolved into n-hexane?	p. 9				
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Were samples extracted by the addition of 40-mL aliquots of hexane/tropolone, vigorous shaking for 3 minutes, waiting 10 minutes for phases to separate, and the removal of the solvent phase?	p. 9				
Was the above extraction process conducted a total of three times for each sample with the three solvent phases being combined?	p. 9				
Were the volumes of the combined extracts reduced to approximately 2 mL of hexane on a rotary evaporator?	p. 9				
When the contents of the flasks were transferred to centrifuge tubes, were the sides of the containers being transferred from rinsed with 2 mL of hexane approximately 10 times, and was that process conducted a total of three times for each container?	p. 9				
If the flasks were set aside for the later collection of column eluents, were they labeled accordingly and rinsed twice with acetone and twice with hexane?	p. 9				
Were any remaining aqueous phases removed from the bottoms of the centrifuge tubes with pipettes and discarded?	p. 9				
Were sample extracts frozen for 1-hour to break up any emulsions?	p. 9				
Prior to derivatization, were frozen sample extracts removed from the freezer one at a time and transferred into another Hexane-prerinsed centrifuge tube so that frozen water and emulsion stayed in the original tube?	p. 10				
Were the original tubes warmed to melt the ice, and the Hexane liberated from the emulsions poured into the second tubes so that the water and emulsion stayed in the original tubes?	p. 10				
(There should be 10-15 mL of extract at this point; if the volume of the extract must be reduced, the nitrogen blowdown of the extract in a 40°C bath is permitted.)	p. 10				
Was the Grignard reagent (Hexylmagnesium Bromide in either Tetrahydrofuran(THF) or ether) warmed to room temperature prior to use?	p. 10				
Was the Grignard reagent not exposed to air during the derivatization process such as by pressurizing its container with nitrogen and expelling it below the surface of the hexane?	p. 10				
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Were tubes agitated with a vortex mixer for 5-10 seconds every 5 minutes for 30 minutes after the addition of the Grignard reagent?	p. 10				
Was 2 mL of HCl added to each sample to neutralize excess Grignard reagent, and the tubes shaken vigorously and vented three times?	p. 10				
Were extract tubes allowed to sit for 30-minutes to allow the hexane phases and the aqueous-HCl phases to separate?	p. 10				
Were the lower aqueous-HCl phases removed from the tubes?	p. 10				
Were extracts blown down with dry Nitrogen in a 40°C bath to approximately 1.5 mL?	p. 10				
Did clean-up columns contain glass wool plugs, Florisil (activated at 110°C), and anhydrous sodium sulfate?	p. 10				
Were clean-up columns rinsed with hexane prior to use?	p. 10				
Were extracts run through columns with the tubes being rinsed three times with hexane into the columns?	p. 10				
Was tetrabutylin (TBT) internal standard added to the sample extracts?	p. 11				
Were sample extracts reduced to analysis volume under nitrogen prior to anlaysis?	p. 11				
Analysis in Sediment Samples					
Were frozen samples thawed prior to extraction?	p.16				
Were portions of samples weighed into centrifuge bottles?	p. 16				
Were the water contents of the samples determined by drying portions of them overnight in a 100°C oven and reweighing until a constant final weight was obtained?	p. 17				
Was pre-extracted sand used as a blank?	p. 17				
When the blanks and the samples were spiked with surrogate, were the walls of the containers avoided and only the sediments spiked?	p. 17				
Was the pH of the deionized water added to each sample and blank adjusted to 2?	p. 17				
Were 100-mL Hexane/0.2%Tropolone aliquots added to each wetted sample aliquot?	p. 17				
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Next, were the samples shaken for one hour on a wrist-action shaker?	p. 17				
Were the sediment and hexane layers allowed to separate for 1 hour?	p. 17				
Were the hexane portions removed from the sediments?	p. 17				
Was the hexane extraction, shaking, and separation repeated once more for each sample?	p. 17				
Was the hexane extract from the second extraction for each sample combined with hexane extract from the first?	p. 17				
Were the volumes of the combined extracts reduced to about 2 mL with a rotary evaporator?	p. 17				
Were the contents of the evaporation flasks transferred to centrifuge tubes with the flasks being rinsed 10 times with hexane?	p. 17				
Were the contents of the centrifuge tubes reduced to approximately 2 mL under streams of dry nitrogen?	p. 17				
Was the granular copper activated with 50/50 HCl until it was bright?	p. 17				
Was activated copper kept under hexane until use?	p. 17				
Was the granular copper rinsed three times with acetone and hexane immediately prior to use?	p. 17				
Were columns rinsed and glass wool plugs rinsed with hexane prior to use?	p. 17				
Were extracts rinsed through columns with hexane?	p. 18				
Were extracts reduced in volume to about 10-15 mL under dry nitrogen?	p. 18				
Was the Grignard reagent (Hexylmagnesium bromide in THF or Ether) warmed to room temperature before derivatization?	p. 18				
Was Grignard reagent transferred to extracts without exposing it to air such as by pressuring container with nitrogen and expelling reagent below the surface of the extract?	p. 18				
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After addition of the Grignard reagent, were extracts vortexed every 5 minutes for 30 minutes?	p. 18				
Was excess Grignard reagent neutralized by the addition of HCl to each sample?	p. 18				
Were samples shaken vigorously and vented three times after the addition of HCl?	p. 18				
Were samples allowed to sit so phases could separate for 30 minutes?	p. 18				
Were the lower aqueous-HCl phases removed from the tubes?	p. 18				
Were extracts blown down with dry Nitrogen in a 40°C bath to approximately 1.5 mL?	p. 19				
Did clean-up columns contain glass wool plugs, Florisil (activated at 110°C), and anhydrous sodium sulfate?	p. 19				
Were clean-up columns rinsed with hexane prior to use?	p. 19				
Were extracts run through columns with the tubes being rinsed three times with hexane into the columns?	p. 19				
Was tetrabutylin (TBT) internal standard added to the sample extracts?	p. 19				
Were sample extracts reduced to analysis volume under nitrogen prior to anlaysis?	p. 19				
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